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INTERIM TECHNICAL PROGRESS REPORT NO. 6

500°C SILICON CARBIDE RECTIFIER PROGRAM

Covering Period 1 January to 31 March 1963

CONTRACT NO. AF 33(657)-7027

ASD TR 7-727(6)

ASD INTERIM REPORT 7-727(6)
March 1963

500°C Silicon Carbide Rectifier Program

Westinghouse Research Laboratories
Beulah Road, Churchill Boro
Pittsburgh 35, Pennsylvania

Interim Technical Progress Report #6
1 January - 31 March 1963

Research Performed under

Contract AF 33(657)-7027
ASD Project 7-727

Wright-Patterson Air Force Base, Ohio

ABSTRACT-SUMMARY
Interim Technical Progress Report

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500°C Silicon Carbide Rectifier Program

H. C. Chang

et al

Westinghouse Electric Corporation

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Epitaxial growth of thin SiC layers of both p and n-type has been achieved on silicon carbide substrates. Small area epitaxial rectifiers are being prepared for evaluation.

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NOTICES

FOREWORD

This Interim Technical Progress Report covers work performed under contract AF 33(657)-7020 from 1 January 1963 to 31 March 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with Semiconductor Department, Westinghouse Electric Corporation, Youngwood, Pennsylvania was initiated under ASD Project 7-727, "500°C Power Rectifier."

Dr. H. C. Chang, Advisory Physicist, Semiconductor Department, Westinghouse Research Laboratories was the Principal Investigator of Phase I of this project. Others who cooperated in the research and in the preparation of the report were: Dr. R. B. Campbell, Senior Physicist, Dr. V. J. Jennings, Senior Chemist, Mr. L. Kroko, Senior Engineer, Mr. J. Ostroski, Senior Chemist; and Mr. D. Barrett, Research Chemist; all members of the Power Device Department of the Westinghouse Research Laboratories.

Your comments are solicited on the potential utilization of the information contained therein as applied to your present or future production programs.

TABLE OF CONTENTS

I. INTRODUCTION

Efforts during this project have been directed toward determining the most feasible method for the production of 1 ampere silicon carbide rectifiers. A detailed production development plan has been drafted and needs only some refinement. Although all one-ampere rectifiers currently fabricated are produced by the sublimation method, the epitaxial growth of doped silicon carbide layers is being studied as an alternate means of junction formation.

In the study of rectifier fabrication emphasis has been placed on stabilization of electrical characteristics at elevated temperatures. As a result of the progress made in the use of ambient atmospheres it is felt that the present encapsulated rectifiers can be operated for up to 1000 hours at 500°C without deterioration of the electrical characteristics. Results of some of these life tests are discussed in this report.

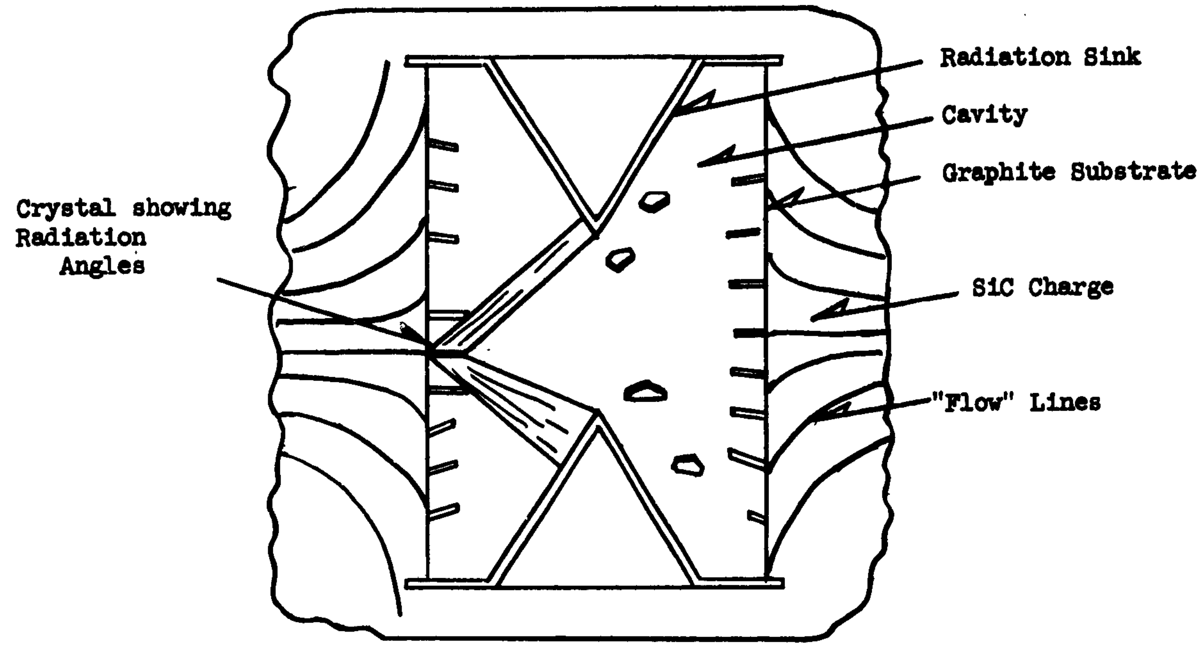
Current efforts in the sublimation method of crystal growth are concerned primarily with controlling nucleation to increase the yield of usable crystals. The experiments performed in this period have yielded important information which lead to a better understanding of the sublimation process.

II. CRYSTAL GROWTH

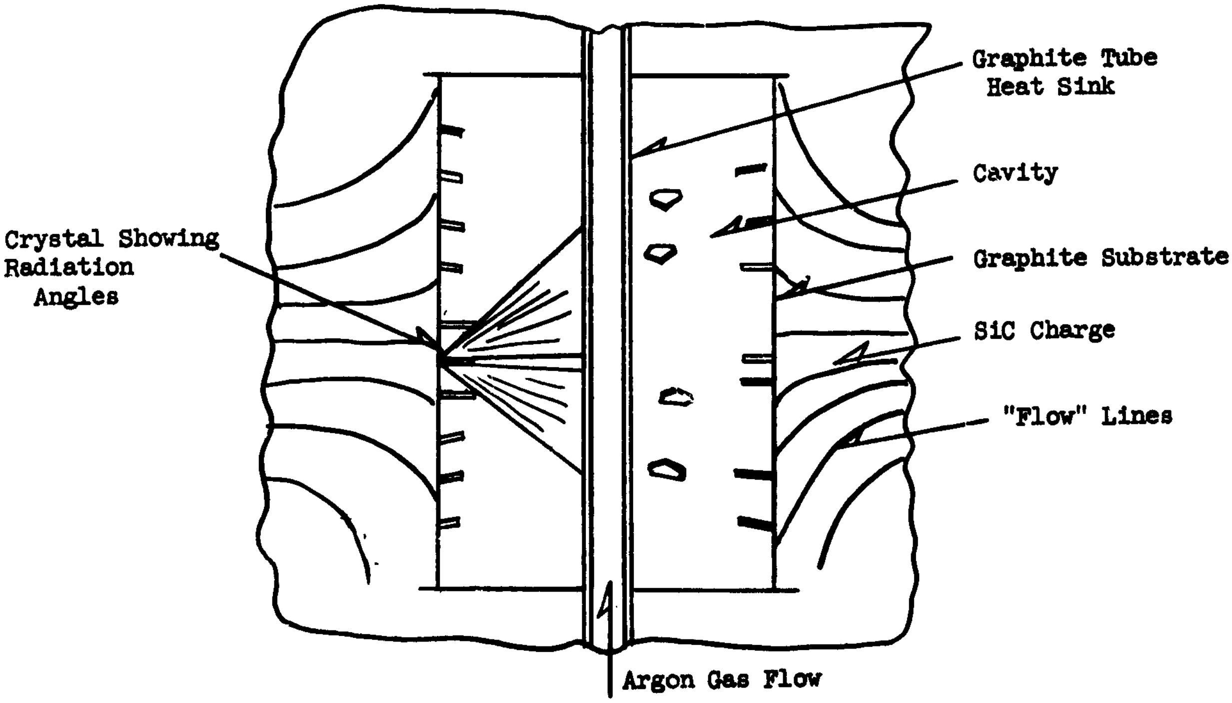
Crystal growth imperfections account for the generally experienced low yield of suitable crystals. The average yield from each preparation in a laboratory-type furnace is about 50 crystals which measure at least 2 mm or larger across the platelet face diagonal. About 13 of these crystals (26%) are large enough and have sufficient crystal perfection to be considered useful for further processing. (A minimum platelet size of about 4 mm is necessary for one ampere device fabrication; smaller crystals are used for lower rated devices i.e. 1/4 amp.) The yield is reduced by the size limitation, but imperfections are the major limitation.

A number of experiments were made to investigate the effect of changes in cavity geometry and radiation sink design. A "normal" cavity is 1 1/4 inch in diameter and 2 inches long. Cavities having a diameter of 2 inches and a length of 2 or 3 inches were used. Since crystals grow within a cavity by radiating the heat of sublimation to a heat sink (the cavity ends). Larger diameter increases the chance for larger numbers of crystals to "see" the cooler heat sink, therefore crystal growth should proceed with increased size. In addition, the increase in substrate surface area should result in a larger number of crystals from each preparation. The schematic drawing shown in Figure 1 illustrates the cavity arrangement and radiation paths to the heat sink.

Two kinds of heat sinks were used with the larger diameter cavities. In the first, an arrangement was used as shown in Figure 1. In the second, the heat sink was changed to a graphite tube through which



Schematic Cavity Arrangement
Figure 1



Schematic Cavity Showing Tube Heat Sink
Figure 2

Argon passed axially through the cavity and charge as shown in Figure 2. In three successive runs the argon flow rate was doubled. Crystals grown in all cases were not significantly larger and were less perfect than crystals grown using the "normal" (i.e. 1 1/4 inch diameter cavity) arrangement. The crystals were "beveled" rather than being flat platelets showing that the thermal geometry about the larger diameter cavity was inadequately controlled even though corrections were applied after each experiment. A larger number of crystals, about twice as many, were grown; but they were imperfect and did not increase the yield.

The length of the cavity was reduced to 2 inches in an attempt to relieve the thermal gradient difficulty but the same result was obtained.

In another trial a massive cone shaped heat sink was located at the cavity end in place of the flow tube. This sink similarly altered the thermal geometry and resulted in growth of "beveled" crystals.

Although the larger cavity and substrate offered an advantage in the number of crystals and availability of heat sinks, the yield was decreased due to altered thermal geometry about the cavity. The results were not conclusive.

These experiments and some others conducted under contract AF 33(657)-8719 also indicated that there is an effective limit of length for the crystals growing from the graphite substrate surface toward the center of the cavity. This indication strongly suggests that under these experimental conditions the degree of supersaturation of the vapor decreases as the crystals extend into the center of the cavity. Some analysis of this problem is being conducted.

In the effort to control the process of nucleation many experiments designed to control the number and spacing of "seeds" have been conducted.

In one experiment a "normal" run of crystal growth was made and then the more undesirable crystals removed from the cavity by breaking them loose from the substrate. Only those crystals which were well oriented and isolated from neighboring crystals were left intact. The charge was returned to the furnace and growth conditions reproduced. Although there was a little growth on some of the crystals, several of the crystals actually decomposed. New growth also occurred on the substrate and on the spots where crystals had been broken from the substrate. The fact that larger crystals could decompose while smaller crystals grew lends some credulance to the idea of rapid decrease in the degree of supersaturation towards the center of the cavity. Probably the the process of opening and closing the charge produced thermal assymmetries not easy to detect nor easy to control. This experiment will be modified and conducted again.

In another experiment several preparations were made in which silicon carbide seeds were implanted on the graphite substrate surface. Green silicon carbide was sieved to 20-48 mesh size and diluted with 10 parts by weight of graphite. A slurry was made using a sugar solution which was painted on the substrate surface. Painted spots (1/2 - 1 cm) were used so that growth could be observed on the normal substrate next to the coated area. The substrate was heated to drive off water and to carbonize the sugar. The normal grown junction preparation technique was

used. The result was an increase in density of growth on the painted spots over those grown on the normal substrate surface as expected but there was also a great deal intergrowth.

Grown junction crystals were prepared during the course of the experiments described above and by several "normal" crystal preparations for processing as rectifier devices.

III. EPITAXIAL GROWTH

The feasibility of growing single crystal layers of silicon carbide on n-and p-type substrates has been demonstrated but no conclusive evidence of rectification has been shown up to the present time.

P-type layers approximately .6 mil thick were grown on n-type substrates using diborane as the dopant. After growing the original crystals were lapped with boron carbide until only a thin n-type substrate and the grown p-type layer remained. A gold-tantalum-aluminum contact is alloyed to the p-type surface and a gold-tantalum contact was alloyed to the n-type surface simultaneously. After etching in molten sodium peroxide the device was electrically tested for rectification. At 50 volts PRV the current was 320 μA; at a forward voltage of 10 volts the forward current was 80 μA.

Other samples are being prepared and will be reported upon in subsequent periods.

IV. DEVICE FABRICATION

During this period work on devices included prolonged tests at 500°C and preliminary tests at -65°C. Rectifier L-43 is presently operating in an ambient of 500°C with a forward current of 1 ampere and a peak reverse voltage of 150 volts.

This rectifier was processed with the usual, helium treatment and encapsulated in a vacuum of 10^{-6} mm Hg. The initial half wave average reverse current at 150 volts PRV and 500°C was 200 μA. After 350 hours of operation at 1 ampere load the reverse current increased to 250 μA. The results are shown in Figure 3. Prior to life testing the unit was cycled 5 times. A cycle consists of 8 hours at 500°C ambient with forward current of 1 ampere and 150 volts PRV and 16 hours (overnight) at room temperature with no load. Cycling did not produce any changes in the electrical characteristics.

Rectifier L-42 was similarly processed and encapsulated. After several cycles (8 hours on, 16 hours off) the temperature of the rectifier was reduced to -65° with dry ice. No electrical tests were made at this temperature. Before cooling, the rectifier showed the following electrical characteristics at 500°C ambient: half wave average reverse current of 250 μA at 150 volts PRV and a forward current of 1 ampere. After cooling, the rectifier was again placed in a 500°C ambient and showed the same electrical characteristics as initially.

Ambient atmosphere test procedures have been modified for processing rectifiers in quantity. In the previous method, single units were tested while under reverse bias. It has been established that a reverse voltage is not

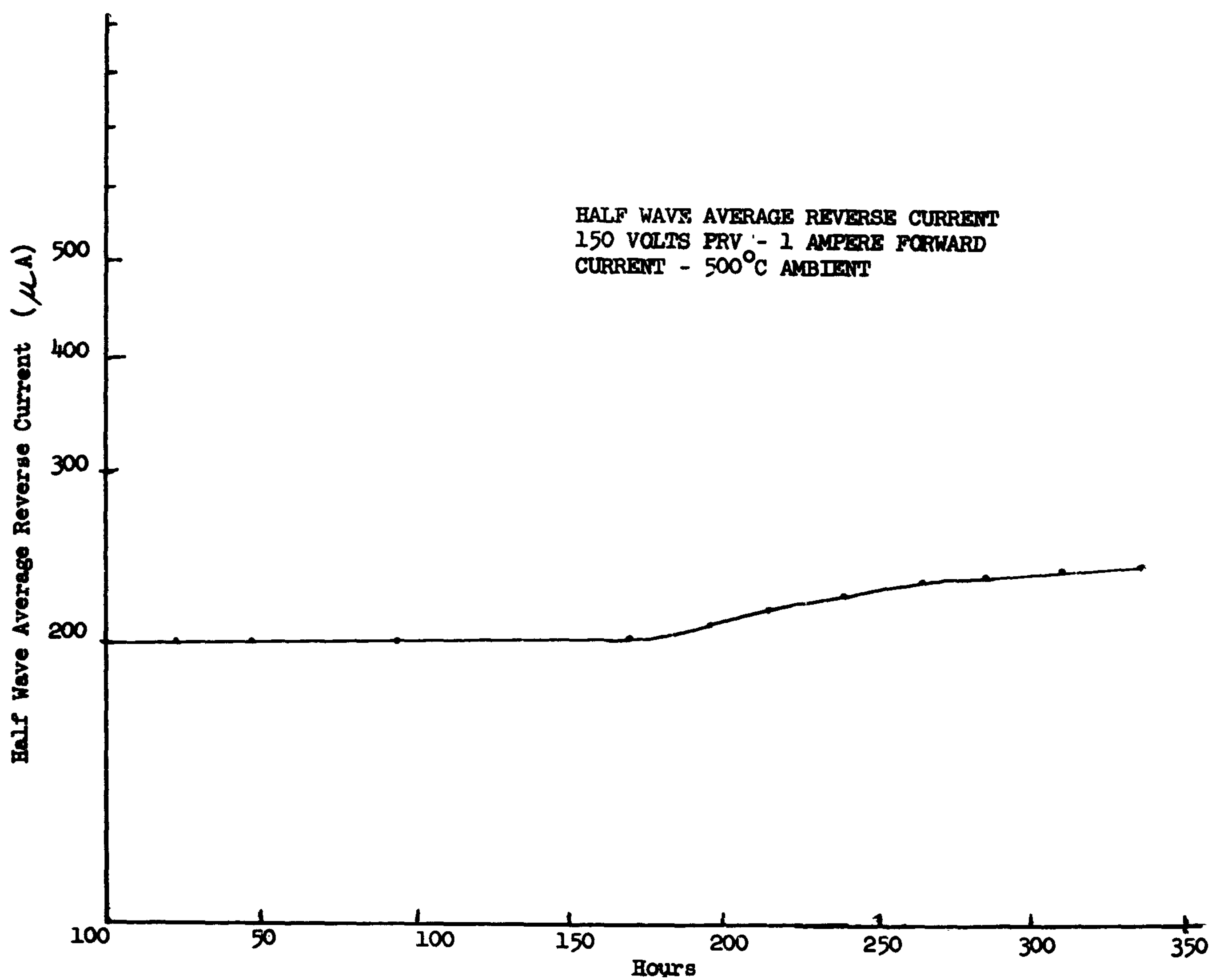

HALF WAVE AVERAGE REVERSE CURRENT
150 VOLTS PRV - 1 AMPERE FORWARD
CURRENT - 500°C AMBIENT
Half Wave Average Reverse Current (μA)
500
400
300
200
100
50
100
150
200
250
300
350
Hours

FIGURE 3

necessary in the reduction and stabilization of the reverse current with helium or water vapor while at 500°C, as shown by the data taken with rectifier L-41.

Rectifier L-41 was processed as usual with gold-tantalum contacts, tungsten backing plates and nickel base. The unit was placed in the atmosphere furnace and evacuated to about 10^{-6} mm Hg. The temperature was then raised to 500°C. The rectifier showed a reverse current of 340μA at 150 volts PRV. The voltage was removed from the rectifier and the quartz tube backfilled to 1/2 an atmosphere with helium for 30 minutes. The system was again evacuated to about 10^{-6} mm Hg and held for 30 minutes. The reverse voltage was again applied to the rectifier and a reduction of reverse current to 50μA at 150 volts PRV was observed. The cycle was repeated to determine if any further reduction in reverse current would occur. The tube furnace was again backfilled to 1/2 an atmosphere of helium while a reverse voltage of 150 volts PRV was applied. The reduced reverse current remained the same at 50μA and no change occurred upon evacuation again. This indicates that many rectifiers could be treated at the same time without requiring electrical leads and test equipment for the application of reverse voltage. Temperature is the important factor in the reduction and stabilization of the surface leakage and the maximum change occurs between 425°C and 500°C.

V. FUTURE PLANS

The use of growth cavity substrates having a different configuration will be initiated to investigate the nucleation phenomena. These experiments are designed to improve the yield of usable crystals.

The study of the conditions necessary for the epitaxial growth of doped layers on SiC substrates which are suitable for rectifier fabrication will continue. Epitaxial junction rectifiers will be made for evaluation.

The fabrication and life and storage tests of 1A rectifiers will continue.

More efficient methods of passivation for quantity production of rectifiers will be investigated.

Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio
Rept. No. 7-727(6). 500°C SILICON CARBIDE RECTIFIER PROGRAM.
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17 pg. incl. graphs. Unclassified Report

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III. Chang, H.C., et al.

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